Texas Natural Resource Conservation Commission

Inspection Checklist for Laboratories Operated by Wastewater Permittees

The purpose of this checklist is to assist TNRCC field investigators who inspect laboratories operated by permittees that provide analyses for self-reporting requirements specified in a wastewater permit. Commercial laboratories that provide analytical services for wastewater permittees are inspected by the Quality Assurance Specialist from the TNRCC Laboratory in Houston.

This checklist is not required to be used at all times when inspecting permittee-operated laboratories. However, it is recommended that the checklist be used as a guide to aid the field investigator in orally reviewing the laboratory's testing procedures and quality control program.

The checklist is set up as follows:

1. Copies of the Appropriate Method References On-hand: This section lists the test procedure references the laboratory should have on hand. A laboratory is not required to have all of those listed, but it must have the ones it is using and referencing at the time of the inspection.

   There are seven references listed and each has a "●" with a number inside it next to the reference. After the reference, there is a blank line where the field investigator can identify which references are on hand.

2. General Laboratory Conditions and Laboratory Instruments, Equipment & Reagents: After each item in these sections, a "●" (one or more with a number inside it) is shown. If the field investigator identifies a problem for any of these items, the number inside the bullet will refer the field investigator and the laboratory to the regulations covering that provision.

3. Laboratory Methods: Prior to conducting the inspection, the field investigator must check the permit and identify the self-reporting parameters for which the laboratory is testing.

   Each test method/question in this section has a "●" (one or more with a number inside it) followed by a blank line. If a discrepancy is identified, the field investigator can note the problem on the line and the number in the bullet will refer the field investigator and the laboratory to the references covering that method.

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Note: If laboratory personnel are interested in laboratory training or need technical assistance, they may contact Terry G. Mills in the TNRCC Laboratory at 281-457-5229.

COMPANY/LABORATORY NAME:_____

PERMIT NO:_____
INSPECTOR:_____
DATE:_____

"*" INDICATES A RECOMMENDED PROCEDURE OR ACTION
"●" with number inside is given as the reference for the specifications listed in each section.

Copies of the Appropriate Method References On-hand:

Standard Methods For the Examination Of Water and Wastewater (Current Edition) _____

TAC Chapter 30, Section 319 "General Regulations Incorporated Into Permits", Subchapter A _____

CFR 29/Part 1910 (current edition) Health & Safety _____

40 CFR/Part 136 (current edition) Protection of Environment _____

EPA methods manual 600/4-79-020 "Methods Of Chemical Analysis Of Water and Wastes" _____

EPA QC methods manual 600/4-79-019 "Handbook For Analytical Quality Control In Water And Wastewater Laboratories" _____

TNRCC 20/40 Hour Laboratory Manual _____

(Copy required if used in lab)

GENERAL LABORATORY CONDITIONS

Laboratory clean and orderly _____

All spills cleaned up immediately _____

All MSDS sheets present and available _____

Acids and bases separated in storage _____

Lab properly ventilated and cooled _____

All lab records kept 3 years _____

All required licenses/certif. posted _____

*****Voluntary "Certified Laboratory Analyst" Certificates posted _____

All containers labeled _____

All chemicals dated rec'd/opened _____

Methods in use are available in lab _____

Emergency eye washes/showers present _____

Fire extinguishes present/accessible _____

LABORATORY INSTRUMENTS, EQUIPMENT, & REAGENTS

"*" indicates a recommended procedure or technique.

Reagents:

American Chemical Society (ACS)/reagent grade or better _____

Glassware:

All class "A" and Borosilicate _____
(unless otherwise indicated by method)

Water:

Potable water supply present _____
Distilled or De-ionized water:
Generated on-site _____
Purchased elsewhere _____
Meets Reagent Grade/Type III
specifications or better _____

Balances:

Serviced by technician annually _____

Service certificate on file _____

Calibrated prior to each use _____

Constant wt. chk. prior to each use _____

Daily log & maint. book kept _____

DO (Dissolved Oxygen) Meter/Probe:

Temp. compensation auto/manual _____

Meter read-out to 0.1 _____

Calibrated prior to use _____

Re-calib. if meter moved _____

Calibrated or standardized against
Winkler daily _____

Winkler titrant normality chk weekly _____

Daily log & maint. book kept _____

pH Meter:

Temp. compensation auto/manual _____

Meter read-out to 0.1 _____

Calib. with 2 buffers prior to use _____

Do buffers bracket pH of samples _____

Re-calib. if meter moved _____

*****Buffer/samples near same temperature _____
(Follow manufacturer's instructions)

Daily log & maint. book kept _____

BOD Incubators:

Interior clean/no spills _____

No light in incubator _____

Temp. at $20^{o}C \pm 1^{o}C$ _____

Temp. checked daily when in use _____

Temp. adjustments noted _____

Temp. measured in H_2O filled btl _____

Daily log & maint. book kept _____

Fecal Coliform Incubators:

Water/interior clean/no spills _____

Temp. checked daily when in use _____

Temp. at $44.5^{o}C \pm 0.2^{o}C$ _____
Temp. adjustments noted _____

Thermometer submerged in H_2O _____

Water level maint. daily _____

Daily log & maint. book kept _____

Ovens/Furnaces:

Interior clean/no spills _____

Temp. checked daily when in use _____

TSS oven at 103^{o}C to 105^{o}C _____
VSS furnace at 550^{o}C ± 50^{o}C _____

Temp. adjustments noted _____

Thermometer in oven when in use _____

Daily log & maint. book kept _____

Refrigerators:

Interior clean/no spills _____

Temp. checked daily when in use _____

Temp. at 4^{o}C (*****± 2^{o}C) _____

Temp. adjustments noted _____

Temp. measured in H_2O filled btl _____

Daily log & maint. book kept _____

Thermometers:

Calib. checked annually _____

All thermometers "NITS" traceable _____
Broken units (Hg) disp. of properly _____
Daily log & maint. book kept _____

LABORATORY METHODS

"*" indicates a recommended procedure or technique.

****NOTE: THE TERM "BATCH" MEANS 10 SAMPLES OR LESS ANALYZED** (EXCEPTION TO ABOVE IS FECAL COLIFORM WHERE A BATCH IS 20 SAMPLES OR LESS)**

Chlorine Residual:

Name/Number of method used _____

FAS normality checked weekly _____

Cl_2 Equiv. Std. each use _____

Sample volume adequate ($\leq$ 500 mL) _____

Each measurement "MnOx" corrected _____

Each measurement "Blank" corrected _____

Daily log & maint. book kept _____

Laboratory BOD Chlorine Neutralization:

Sodium Sulfite used _____

Sodium Sulfite prepared fresh daily _____

Sample volume adequate ($\leq$ 2 liter) _____

Daily log & maint. book kept _____

BOD_5 (Biochemical Oxygen Demand) at 20°C ± 1°C

Name/Number of method used _____

Samples at **20°C ± 1°C** when analyzed _____

Dilution water at **20°C ± 1°C** when used _____

Controls

Dilution Water Blanks

Dilution water blank with each day _____
(*****2 recommended)

Dilution water @ 20°C ± 1°C prior to use _____

Depletion ≯ 0.2 mg/L _____

Nutrients:

In-house nutrients used _____

Commercially prepared used _____

If "yes", is certification on file _____

Reagents dated Rec'd/Opened _____

Seed Controls

Seed controls with each day _____

Prepared fresh daily _____

1 control used (required) _____
(*****3 Recommended at 5%, 10%, 15%)

40% to 70% DO depletion in control _____

Seed Corr formula [(B1 - B2) x F] _____

Seed Corr ≮ 0.6 mg/L and ≯ 1.0 mg/L _____
(***** .5 to 1.1 acceptable on occasion)

If commercial seed used: _____

Certification on file _____

Used immed. after aeration _____

Reagents dated Rec'd/Opened _____

Prepared fresh daily _____

If in-house (raw/influent) seed used: _____
BOD tested on in-house seed _____
Toxic effects observed _____
pH adjusted 6.5 to 7.5 _____
Used immed. after aeration _____
Prepared fresh daily _____

Glucose/Glutamic Acid Standards (GGA's)

GGA's set up each day _____

GGA prepared fresh daily _____

Oven dried/cooled desic. prior to wt. _____

GGA set up as duplicates of 2% conc. _____

GGA BOD = 198 mg/L ± 30.5 mg/L _____

BOD SAMPLES

48 hr. holding time observed _____

Sx @ 20°C ± 1°C prior to analysis _____

Sample volume adequate ($\leq$ 2 liter) _____

At least 3 different conc. used _____

All particle sizes < 3 mm (homogenized) _____

If Sx homogenized, what device is used _____

if particle sizes > 3 mm _____

BOD ***SAMPLE*** pipet tips $\leq$ 3 mm bore _____

Was sample seeded if:
Sample not analyzed within 2 hrs. _____
Sample temperature changed _____
pH adjusted to 6.5 - 7.5 _____
Sample disinfected _____

BOD btl's have ground glass stoppers _____

Distilled water seal on bottles _____
(***Dilution*** water seal recommended)

*****BOD verified with BOD/VSS ratios _____

True duplicate test performed _____
1 dup per 10 or per batch _____
Mean, WL, & CL charted _____

Reportable BOD calculated _____
5 day DO ≥ 1.0 mg/L _____
Corrected depl. ≥ 2.0 mg/L _____
Statistical analysis used _____
Toxic effects indicated _____

Daily log & maint. book kept _____

Carbonaceous BOD_5 @ 20ºC ± 1ºC

TCMP inhibitor used _____

Sample volume adequate (≮ 2 liter) _____

All controls with inhibitor added _____

TCMP added to all sample dilutions _____

All above rules for BOD applied _____

Daily log & maint. book kept _____

Fecal Coliforms

Name/Number of method used _____

Sx Container pre-sterilized with
Sodium Thio. in container _____

Sample volume adequate (≮ 125 mL) _____

6 hour holding time observed _____

Sterilized grad cyl/pipets used _____

Culture media prepared fresh _____

Commercially prepared media used _____
(If "Yes", documentation on file) _____

Commercial sterile culture plates used _____
(If "Yes", documentation on file) _____

Commercial sterile filters used _____
(If "Yes", documentation on file) _____

0.1 mL dilutions done in serial dilu's _____

Sterile buffer wash water used _____

Apparatus sterilized between each use _____
3 different dilutions used _____

All plates/dilutions counted _____

Blank analyzed daily _____

If more than two analysts performing the test,
are both analysts counting
plates at least once per month _____

Is Presumptive & Confirmed QC using
MPN being done once per month on
positive & negative cultures _____

True duplicate test performed _____
1 dup per 5% of tests _____
Mean, WL, & CL charted _____

Daily log & maint. book kept _____

TSS/VSS:

Name/Number of method used _____

7 Day holding time observed _____

Blank analyzed _____
(*****500 mL recommended)

*****Standard Used (Celite or other) _____

Sample volume adequate ($\nless$ 500 mL) _____

Filter used with wrinkled side up _____
("N/A" if no wrinkled side present)

Filter treated prior to use _____

All dried/cooled until at constant wt. _____
± 0.5 mg 1st wt. to 2nd wt. _____

Volume of sample filtered achieves:
≥ 1 mg wt increase over tare wt _____
< 200 mg increase over tare wt _____

Grad. cylinder rinsed properly _____
cyl. 3 times-each through filter _____
funnel walls-3 rinses _____
filter-3 rinses _____

True duplicate test performed _____
1 dup per batch _____
Mean, WL, & CL charted _____
All QC > CL re-analyzed _____

If VSS analyzed: _____
Filter ashed at 500°C ± 50°C prior to use _____

All VSS values ≤ TSS values _____

True duplicate test performed _____
1 dup per batch _____
Mean, WL, & CL charted _____

All QC > CL re-analyzed _____

Daily log & maint. book kept _____

pH @ 25ºC (Laboratory)

Name/Number of method used _____

BOD sample pH measure @ **20ºC** _____

pH meter approved for self reporting
(See "Laboratory Instruments, Equipment,
& Reagents" above for spec's) _____

All pH values within calib. range _____

Sample volume adequate (≯ 50 mL) _____

True duplicate test performed _____
1 dup per batch _____
Mean, WL, & CL charted _____
All QC > CL re-analyzed _____

Daily log & maint. book kept _____

DO (Winkler Titration)

Name/Number of method used _____

Sample collected in 300 mL BOD bottle _____

If volume titrated is 200 mL: _____
Is volume adj to 201.3 mL: _____
Titrant Normality = 0.025N _____

If volume titrated 300 mL: _____
Titrant Normality = 0.0375N _____

If sodium thio titrant purchased: _____
Normality checked weekly: _____

Sodium Thio titrant prepared from stock: _____

Thio. Normality check weekly: _____

Daily log & maint. book kept _____

Ammonia Nitrogen (NH_3-N)

Name/Number of method used _____

Holding times observed as follows:
- Non-preserved analyze immediately _____
  (**WITHIN 15 MINUTES****)**
- Preserved analyze within 28 days _____

Sample volume adequate ($\prec$ 1 Liter) _____

Sample distilled prior to analysis _____

Chlorine neutralized before distill. _____

pH neutralized to pH 9.5 before distill. _____

Distillation apparatus all glass _____

Ammonia free distilled water used _____

If specific ion probe used:
- Sample not distilled _____
- distilled/non-distilled comparison in files _____

Comparison performed annually _____
(or if meter/probe repaired or replaced)

Blank and standards:
- Analyzed with each batch _____
- Distilled with samples _____

True dup/spk tests performed _____
- 1 dup/spk per batch _____
- Mean, WL, & CL charted _____
- All QC > CL re-analyzed _____

Daily log & maint. book kept _____

TKN (Total Nitrogen)

Name/Number of method used _____

Digestion apparatus all glass _____

TKN values **always** ≥ Ammonia _____

Proc. for "**Ammonia Nitrogen (NH_3-N)**"
followed after digestion _____

O&G (Oil and Grease, Total Recoverable)

Name/Number of method used _____

Sample volume adequate (≮ 1 Liter) _____

Sample preserved with H_2SO_4 _____

28 day holding time observed _____

Blank analyzed each day O&G tested _____

*****Blank value ≯ half of MDL _____

Field Dups **OR** Stds each batch _____

Type of standard material used _____

Widemouth, glass jar with TF liner _____

Jar/liner rinsed with freon before use _____
All are grab samples-NO composites _____

Daily log & maint. book kept _____

COD (Chemical Oxygen Demand)

Name/Number of method used _____
Open Reflux: _____
Closed Reflux: _____

Sample preserved with H_2SO_4 _____

Sample volume adequate ($\leq$ 1 Liter) _____

28 day holding time observed _____

Blank and standard each batch _____

Dup and Spk each batch _____

Cl^- < 2000 mg/L per sample _____
Cl^- levels not measured _____
Method used to measure Cl^- _____

*****COD/TOC ratio used to verify COD _____

Daily log & maint. book kept _____

TOC (Total Organic Carbon)

Name/Number of method used _____

Sample preserved with H_2SO_4 _____

Sample volume adequate ($\leq$ 250 mL) _____

28 day holding time observed _____

Blank and standard each batch _____

Dup and Spk each batch _____

Daily log & maint. book kept _____

Phenol (Total Recoverable)

Name/Number of method used * _____
(* use 14th Edition **Only**)

Sample preserved with H_2SO_4 _____

Widemouth, glass jar with TF liner _____

Use brown glass or store in dark _____

Jar/liner rinsed with freon before use _____

Sample volume adequate ($\lessdot$ 1 Liter) _____

28 day holding time observed _____

If Cl_2 present, FAS added _____

Sample/blk/std all distilled _____

Blank and standard each batch _____

Dup each batch _____

*****Spk with batch _____

Chloroform extracts filtered _____

All grab samples-NO composites _____

Daily log & maint. book kept _____

AA (METALS ANALYSIS)

Name/Number of method used _____

If SW846 used, that Method # _____

Sample volume adequate ($\lessdot$ 1 Liter) _____

Sample preserved with HNO_3 _____
Is plastic container used _____
Is **clear**, glass jar used _____
If "No", what type jar _____

6 mth holding time observed (excpt. Hg) _____

Hg holding time 28 days (sol/liq) _____
Cr^6 not pres/run within 24 hrs _____

Blank and standard each batch _____

Sample/blk/std all digested _____

Dup and spk each batch _____

Standard curve/calib. check daily _____

Daily log & maint. book kept _____